YEARLY PROGRESS REPORT

Project Title: Continuous Severe Plastic Deformation Processing of Aluminum Alloys

Covering Period: February 21, 2001 through February 20, 2002

Date of Report: May 20, 2002

Recipient: Wright State University
3640 Colonel Glenn Highway
Dayton, Ohio 45435

Award Number: DE-FC07-01ID14022

Subcontractors: Intercontinental Manufacturing (IMCO), Garland, Texas
Edison Materials Technology Center (EMTEC), Kettering, Ohio

Other Partners: Oak Ridge National Laboratory (ORNL)

Contact(s): PI: Raghavan Srinivasan (Wright State University)
(937) 775-5093, rsrin@cs.wright.edu
Prabir Chaudhury (Intercontinental Manufacturing)
(972) 276-5131, chaudhuryp@imco-bergman.com
Srinath Viswanathan (Oak Ridge National Laboratory)
(865) 576-9917, viswanathans@ornl.gov
David Swenson and Percy Gros (Edison Materials Technology Center)
(937) 259-1365, dswenson@emtec.org, pgros@emtec.org

Project Team: John A. Yankeelov, Elizabeth E. Dahl

Project Objectives:

- Demonstrate that a continuous severe plastic deformation process produces ultrafine-grained stock material of substantial length and diameter (≥30 mm or~1.25 inch) at a reasonable cost.
- Demonstrate that ultrafine-grained material results in energy and cost savings during forging
- Transfer the technology to commercial practice.

Background:

Ultrafine-grained material allows the design and manufacture of aluminum components that use less metal and require fewer manufacturing steps. This provides energy and manufacturing cost savings. Several techniques for producing ultrafine-grained materials are currently being investigated. These techniques are limited in their ability to produce the size and quantities of material needed for commercial use. One technique to produce ultrafine-grained materials by severe plastic deformation is using Equal Channel Angular Extrusion/Pressing (ECAE/P). This technique is a multi-step batch process that produces small cross-section, short length stock, which severely limits its commercialization.

In this program, a Continuous Severe Plastic Deformation (CSPD) process is being developed through simulation and analyses work at Wright State University (WSU) and prototype equipment development at Oak Ridge National Laboratory (ORNL). In addition, Intercontinental Manufacturing Co. (IMCO) is using an experimental approach to establish the processing steps to create UFG aluminum alloy stock materials and to forge these materials into industrial parts with increasing size and complexity. IMCO is producing the industrial size UFG forging stock and demonstrating the usefulness of the material in reducing cost and energy consumption during forging.

Based on the results obtained in this program, a decision was made to file for intellectual property protection. Because of the proprietary nature of the work on the CSPD process, details are not being presented in this report.

Status:

Significant technical accomplishments

1. Task-1 (Demonstration of the CSPD process)

1.1. Analytical modeling and computer simulation

Three different approaches to the CSPD process were identified. Computer simulation using a commercial metal deformation simulation code, and analytical models of the approaches indicate that all three approaches are potentially viable.

1.2. Development of a CSPD machine

1.2.1 In an effort to put together a CSPD apparatus in the shortest time and cost, a decision was made to use, as far as possible, existing equipment at Oak Ridge National Laboratory (ORNL). An inventory and review of all the available processing equipment was conducted. Based on the review, four possible pieces of equipment were initially selected, and after further review, two pieces of equipment were selected for development of the CSPD apparatus. First, the load limits for the selected equipment were measured. The measurements represented the maximum force that could be exerted by the equipment and this information was used in the design of the CSPD die. A 10,000 lb load cell was used for force measurements up to 10,000 lb and a 50,000 lb load cell was used for force measurements over 10,000 lb. Forces ranging from 7,850 to 15,050 lb were measured.

Based on the measured forces and computer simulation of the CSPD process, it was decided to initially limit the sample size to a half-inch cross-section. The 0.5 in. sample is more likely to be compatible with the available equipment. Also, the 0.5 in. die will allow us to carry out a proof of principle experiment, evaluate fixturing, and validate the results of the computer modeling.

Based on the ECAE experiments at IMCO and WSU, and the computer modeling studies at WSU, a CSPD die has been designed. The die will initially allow sample cross-sections of 0.5 in, but the die inserts can be changed to allow larger sample cross-sections up to 1.125 in. Detailed drawings of the die blocks and inserts have been completed and the die is being fabricated. The delivery date for the die is tentatively May 1, 2002. Tryouts of this system will be during the second year of the project.

1.2.2 WSU is developing second design for CSPD. This design will initially use a model material to demonstrate the approach.

2. Task-2 (Demonstration of cost and energy savings)

Since the CSPD process will not be producing material until at least the middle of the second year of this project, the demonstration of the potential cost and energy savings through the use of ultrafine-grained material is initially through the use of stock material produced by ECAE.

2.1. Alloy Selection

Based on the literature data on aluminum alloys and the type of alloys forged at IMCO, initially two alloys were selected for this program. The alloys were AA 6061 and AA 2014. AA 6061 was acquired by WSU and AA 2014 alloy was donated by Kaiser Aluminum for ECAE/P processing. However, after further review of the most recent data on 2XXX series alloys, the final selection of AA 6061 was made.

2.2. Characterization of ECAE/P ½-inch square AA 6061-O

Samples of ½-inch square AA 6061-O were processed by ECAE at WSU using a 120° channel die for up to 6 passes using route B_C. The microstructures of these samples were examined to study the effect of deformation on the precipitates. Samples were also tested in isothermal compression at constant strain rate to understand the deformation behavior of ECAE processed material.

2.2.1 Hardness variation of AA 6061-O

Upon annealing AA 6061 to the O condition (500°C for 1 hr, furnace cool) the hardness of the alloy is $37R_F$ (Rockwell F scale). With successive passes through the ECAE die, the material work hardens to $70R_F$ at a total true strain of 4. This is shown in Figure 1.

2.2.2 Microstructure of ECAE processed AA 6061-O

Figure 2 shows the unetched microstructure of AA 6061 in the as-received (T6 condition) and the annealed (O condition), as well as after 6 passes. In the T6 condition there are a relatively few precipitates visible under optical microscopy since most of the strengthening precipitates are very small and cannot be seen with visible light. Upon annealing to the O condition, the number of visible precipitates increases substantially. After 6 passes, there is a further increase in the number of precipitate particles, probably because of the break up the particles during ECAE. Table 1 summarizes these results.

2.2.3 Flow curves for ECAE processed AA 6061-O

Figure 3 shows the flow curves for the ECAE processed (6 passes) AA-6061-O alloy at constant strain rates of 0.01 and $0.1s^{-1}$. At a strain rate of $0.01s^{-1}$ the flow curves are relatively smooth. There is some work hardening observed at all

Table 1: Change in particles visible under optical microscopy with processing

Condition	% Area of Particles	Particle density (# per mm^2)
As-received T6	1.87	1.1×10^4
Annealed (Transverse)	3.17	2.6×10^4
Annealed (Longitudinal)	3.04	2.6×10^4
6-pass (X)	4.68	3.7×10^4
6-pass (Y)	5.04	3.5×10^4
6-pass (Z)	4.05	2.4×10^4

temperatures and the level of stress decreases with increasing temperature. At a strain rate of $0.1s^{-1}$ there are distinct fluctuations in the flow curve. The frequency of the fluctuations increases as deformation temperature is increased. The cause is still under investigation and may be related to some dynamic processes occurring in the material.

2.3. ECAE/P of 2-inch square AA 6061

Ten 2-inch square billets of AA 6061-O were machined. These billets were processed by ECAE/P for up to 4 passes at the Air Force Research Laboratory facility, Wright Patterson AFB. Six pieces of UFG AA 6061, two after 4 passes and four after 3 passes of ECAE/P, were supplied to IMCO for forging experiments.

2.4. UFG ZK60 Alloy

Because of the early delays in the ECAE/P processing of the AA 6061, additional UFG material was acquired from a separate DOE program (DE-AC05-00OR22725). Three 22 mm x 22 mm x 100 mm billets of UFG ZK60 processed by ECAE/P were obtained for the initial forging trials.

As received (before ECAE/P processing, 2-pass, and 4-pass) ECAE/P materials of ZK60 alloy obtained from the DOE program were characterized for the microstructure. The results of the characteristic microstructures are shown in Figure 4. Microstructures generally show a duplex size variation: uniformly distributed large grains surrounded by very fine grains. The fine grains in as-extruded condition (8.75 μm) exhibit further refinement after 2 (5.0 μm) and 4 passes (4.65 μm) of ECAE/P. The large grains are of the order of 30 – 40 μm, and do not exhibit significant refinement after four passes of ECAE/P.

2.5. Forging of UFG Material

2.5.1 ZK-60

UFG ZK60A processed by ECAE/P was first forged to better understand the production floor adjustments needed for lower temperature forging with UFG materials. A small trial part was selected based on the size of the stock material available and the alloy being forged. This part, shown in Figure 6, is normally forged using a 32 mm diameter and 82 mm long cylindrical extruded stock. However, the UFG ZK60 alloy forging stock used was 22 mm x 22 mm x 95 mm in size utilizing

70% of the stock size. Six parts were forged with extruded stock (0-pass) and UFG (2- and 4-pass ECAE/P) material. The material and forging conditions are listed in Table 2 for comparison. This table, along with the Figure 5, where Forging Load vs. Temperature is plotted, shows that it is possible to decrease the forging temperature of ZK60 by 100°F using a finer grain material without increasing the forging load. Also, the use of smaller stock volume is possible. However, the results are not dramatic in this alloy system because of the refinement of grain size achieved by ECAE/P process is not extensive and the grains are not uniform in size; there are large grains in the microstructure. While the fine grains enhance the superplastic behavior during hot forming, the large grains tend to inhibit the grain boundary sliding locally affecting the total strain contribution. This effect is expected to be much higher in aluminum where the grain refinement can be extensive and would show much better flow at lower temperature.

Table 2: Forging conditions of initial trails with < 25 mm square cross section billets produced by ECAE/P

Forging #	Material	Stock Temp (F)	Die Temp (F)	Max Load (tons)
1	CG ZK60	750	600	123
2	CG ZK60	700	600	300
3	CG ZK60	650	600	203
4	UFGZK60-4Pass	650	600	252
5	UFGZK60-4Pass	600	600	252
6	UFGZK60-2Pass	650	600	219

2.5.2 AA 6061

The second set of forging trials were conducted using larger size billets and correspondingly larger parts using the selected aluminum alloy AA 6061. The UFG AA 6061 was produced at Air Force Research Laboratory (AFRL) using 3 or 4 pass ECAE/P process. Table 3 shows the forging trials with two different dies and the variables for these forgings. Normally AA 6061 alloy is forged at around 915°F, however, these parts were forged at progressively lower temperatures from 780°F to 600°F. In order to avoid grain coarsening, the heat up time before hot forging was limited to 30 minutes. The UFG AA 6061 stock material exhibited up to 300°F lower forging temperature (Figure 7) and use of 15% less forging stock (Figure 8). The results of the forging trial show that the potential for energy savings is very high with UFG AA 6061 alloy. The lower temperature of forging, shorter time of heating, and use of less stock material contribute significantly to the energy savings.

The above forging trials were conducted with few very small size parts. In order to understand and determine the potential for energy savings across the forging industry, it is important that similar trials are performed with larger parts. However, the current technology and capability in ECAE/P does not allow such trials without producing large size UFG stock. The next section describes the accomplishments in the first year in producing such material for further forging trials for forging larger parts.

Table 3: Forging conditions of initial trials with > 25 mm but < 50 mm square cross section billets produced by ECAE/P

Forging No.	ECAE Pass	Operation	Die Temp,(ºF)	Stock Heat up Time, (min)	Stock Temp, (ºF)	Hardness	Comments
1-2250-1B	4	Flatten to 1"/ Finish to .62"	760	30	760	N/A	
		Machine Sand & Handwork	Done	Done	Done	19.7 HR_E	
2-2250-1B	4	Flatten to 1"/ Finish to .62"	660	35	680	N/A	
		Machine Sand & Handwork	Done	Done	Done	18.80 HR_E	
3-2250-1C	4	Flatten to 1"/ Finish to .62"	760	30	780	N/A	
		Machine Sand & Handwork	Done	Done	Done	22.60 HR_E	
4-2250-1C	4	Flatten to 1"/ Finish to .62"	660	35	700	N/A	
		Machine Sand & Handwork	Done	Done	Done	19.50 HR_E	
5-1774-1B	3	Flatten stock to 1"	600	30	680	N/A	
		Preform to .032" shim / Finish Forge	660	30	600	16.50 HR_E	Normal starting stock size
6-1774-2C	3	Flatten stock to 1"	600	30	740	N/A	
		Preform to .032" shim / Finish Forge	640	40	700	14.00 HR_E	15% less starting stock

2.6. Large Size UFG Material Production

Based on the original plan as proposed in this program, an ECAE/P die, that would produce 100 mm x 100 mm cross section billets, was designed and machined at IMCO. An analysis of the ECAE/P process using the die was simulated and the die design was refined on the basis of the simulations. The drawing of the die is shown in Figure 9. This die was placed under a 2000-ton hydraulic press and 100 mm x 100 mm x 457 mm billets were processed through the die. After making one pass each on five billets, one of the inserts fractured during the trial. The weaknesses in the design of the die support and the insert are being corrected, and the UFG AA 6061 will be produced for forging trials in the second year.

2.7. Estimations of Gas Consumption and Energy Savings

2.7.1 Gas Consumption

In order to determine the cost and energy savings, the gas consumption studies were conducted. This study included the measurement of gas consumption to attain various temperatures. This study was conducted in two steps. First, different sizes of forging stocks (slugs) from 1 inch to 8 inch in diameter were heated up in a production stock oven. Each slug had a thermocouple inserted in it to its mid diameter to monitor the temperature during heat up and soak time. The temperature variation as a function of time for each size of slug is shown in Figure 10. The time required for larger slugs to reach the set temperature was longer as expected. The gas consumption during the ramp up as well as for the steady state heating of stock was also measured in this study. The results of this study are shown in Figure 11. Figure 11 shows the gas usage as a function of forging temperature for three sizes of the stock material. Larger the stock size and higher the temperature, greater is the amount of gas consumed. The analysis shown here shows an example of gas consumption model for the stock oven studied.

Second part of the study was to record gas consumption for production loads in various production stock ovens. The preliminary results are shown in Figure 12. The results show that the gas consumptions in the stock ovens primarily depend on the temperature and time of operation. The size of the load also has a contribution in the gas consumption, although small because of the loss of heat through the doors that are opened frequently during the forging operation. However, this part of the investigation will be refined after further study of the production runs.

2.7.2 Energy Savings Estimations

The first year of the work in this program showed that there are five primary sources of energy savings when ultrafine-grained forging stock is used in forging industrial parts. They are:

- ▷ Lower forging temperature
- ▷ Shorter heat up time
- ▷ Smaller forging stock size
- ▷ Fewer number of hits
- ▷ Lower forging load.

The ability of the of UFG material produced by the CSPD process is expected to allow hot forging process to use less energy through reduced heat up time and lower forging temperature. As per Figure 11, the estimate for AA 6061 shows that there is approximately 15% reduction in gas consumption per 100°F reduction of forging temperature. Because of the lower forging temperature the heat up time reduces considerably and for every 100°F decrease in forging temperature the heat up time decreases approximately one to two hours depending on the stock size. Assuming a stock size of 6 inch diameter (average of all forgings), the potential gas savings for forging AA 6061 part is estimated to be as high as 40 %.

As shown in Figure 8, the size of the forging stock can be reduced by 15% when UFG AA 6061 was used in small forgings because of the better flow of the material inside the die. This reduction in forging stock size translates into major energy savings related to aluminum forging stock production as well as reduction in forging flash that is normally recycled. The cost and energy savings associated with a 15% reduction in stock material would depend upon whether the reference values are for production of “new” aluminum or for recycled stock.

Additional cost savings can be achieved when a fewer number of hits is used because of the better forgeability of the UFG material. Elimination of one in three hits can reduce the forging energy consumption by 33 %. Other important energy saving has been shown to be because of lower forging load (Figure 6). Lower forging load requires less energy for forging. The full impact of this aspect of energy savings will be realized if it becomes possible to use a smaller forging press.

3. Task-3 (Transfer of technology)

3.1. IMCO and EMTEC have started exploring potential commercial applications.

3.2. In addition to contacts with individual companies, presentations on the project have been made at the following forums:

- ▷ NADCA -Cincinnati (Nov. 1-2, 2001)
- ▷ ASM-Indianapolis (Nov. 5-7, 2001)
- ▷ Dayton industrial exhibit -Dayton (Nov. 7-9, 2001)
- ▷ EMTEC TSC -Dayton (Nov. 14, 2001)

3.3. Plans are being made to determine potential routes and sources for funding for commercialization, such as the Advanced Technology Program (ATP).

- ▷ At the quarterly review meeting held February 7-8, 2002, EMTEC presented to the group their prior experience in winning an ATP grant and outlined a timeline for developing a proposal for a continuation of this program.

3.4. A website for the project is under development. The site will be maintained by EMTEC and will be used to disseminate non-proprietary information, such as the content of this report, as it becomes available.

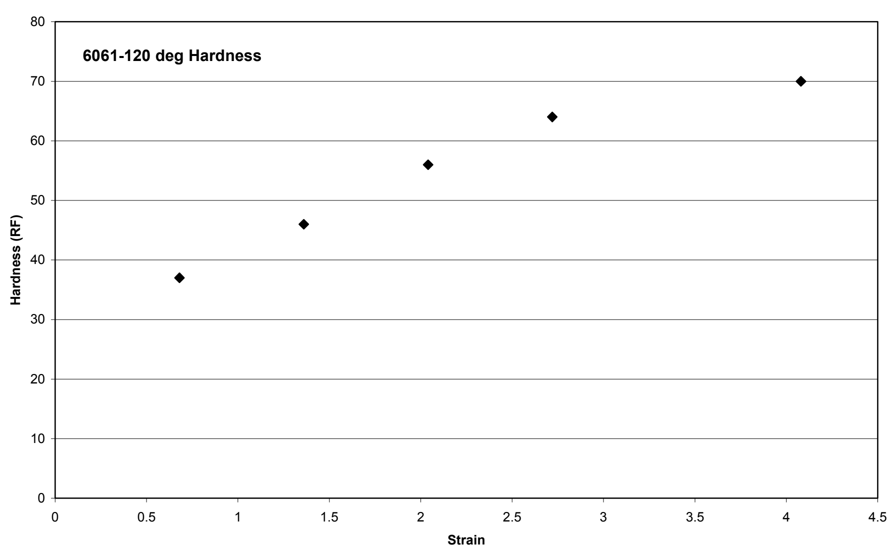


Figure 1: Variation of hardness of AA 6061-O with multiple passes through a 120° ECAE die

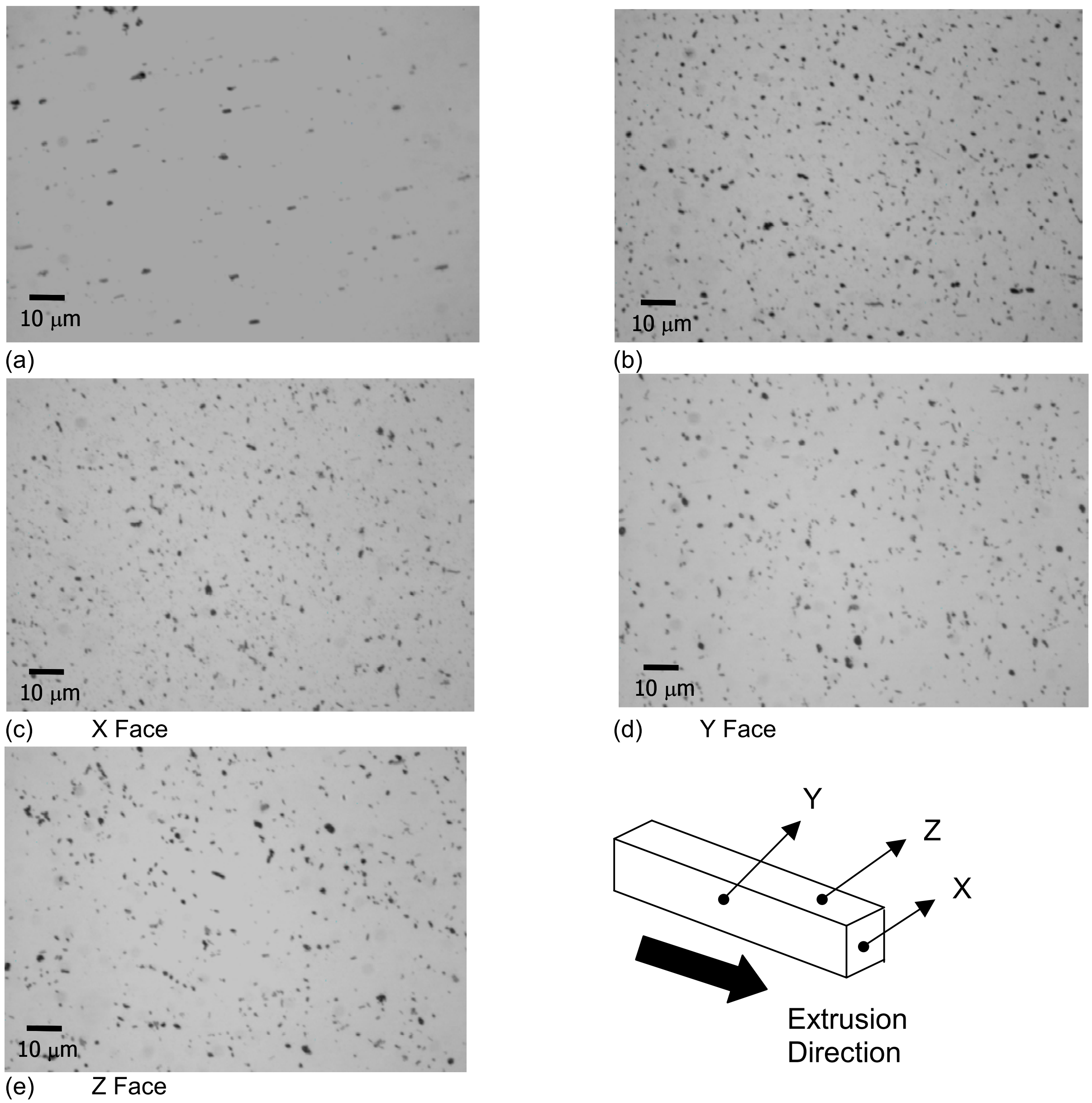


Figure 2: Microstructure of AA 6061 (a) as-received AA 6061-T6 (b) AA-6061-O after annealing (500°C 1hr, FC), (c-e) AA 6060-O after 6 passes through a 120° ECAE die

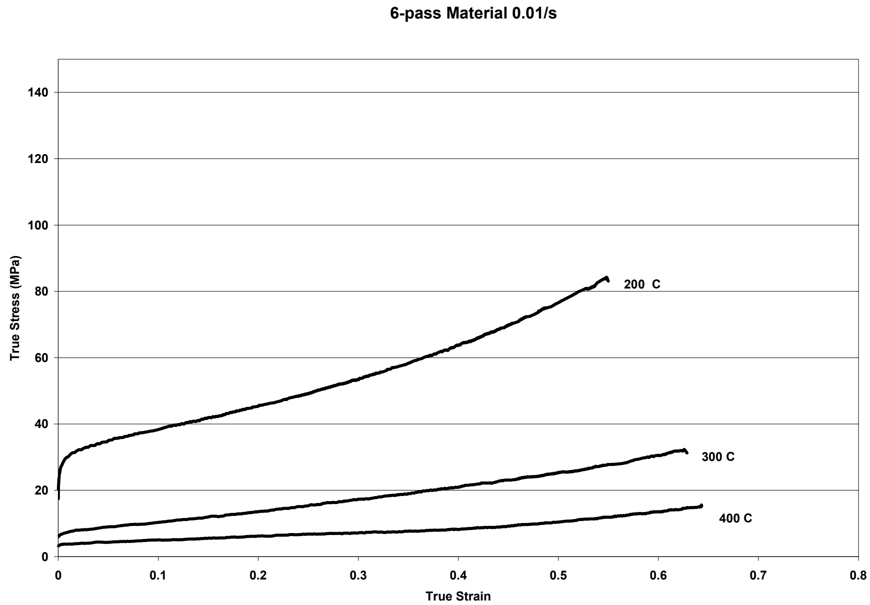


Figure 3(a): Flow curves for AA 6061-O after 6 passes through a 120° ECAE die at $\dot{\varepsilon} = 0.01 s^{-1}$

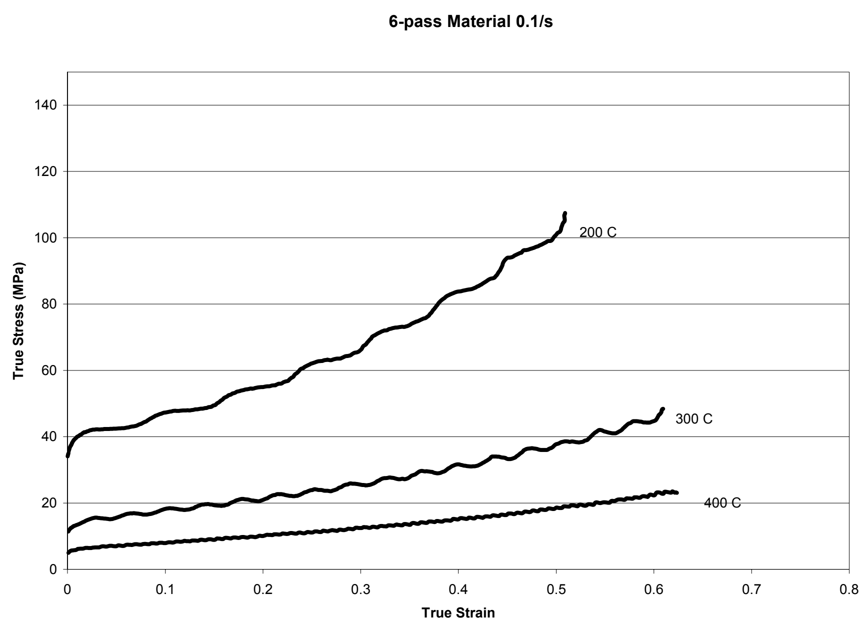


Figure 3(b): Flow curves for AA 6061-O after 6 passes through a 120° ECAE die at $\dot{\varepsilon} = 0.1 s^{-1}$

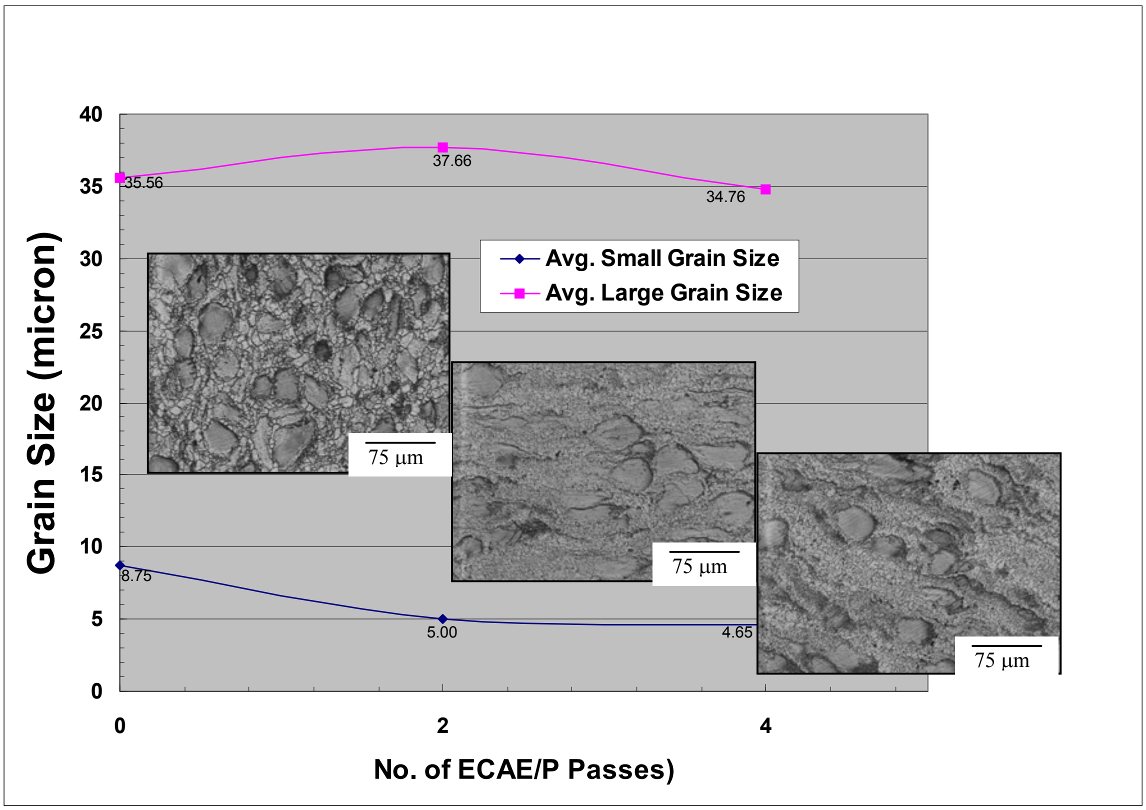


Figure 4: Variation in grain size and microstructure after ECAE/P processing of ZK60 at 260°C.

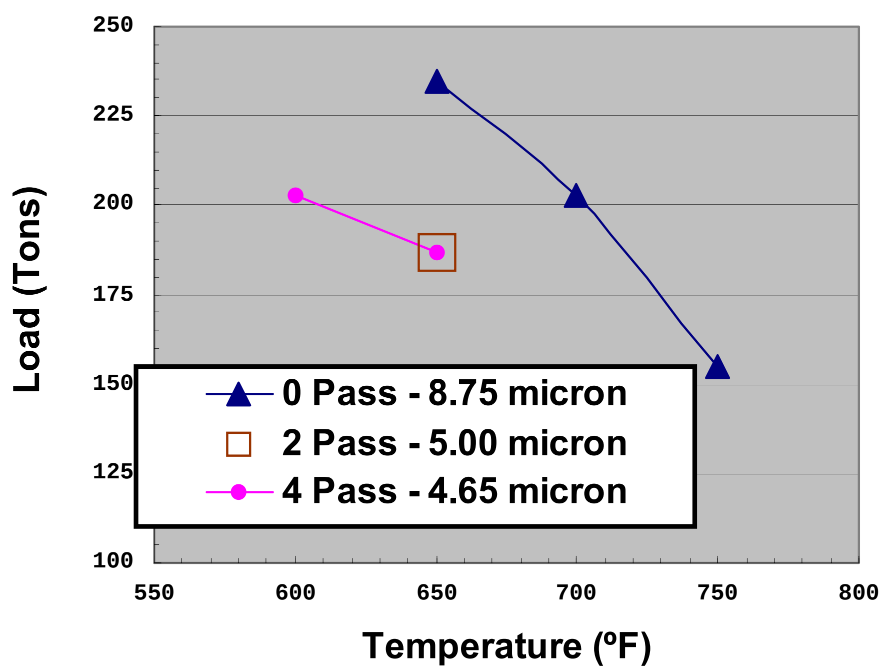
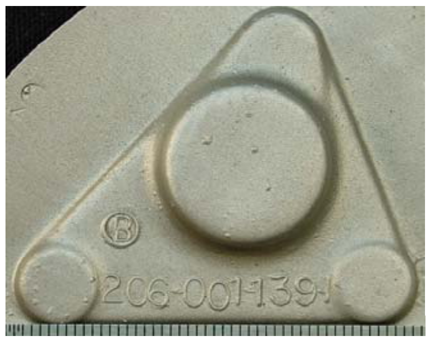
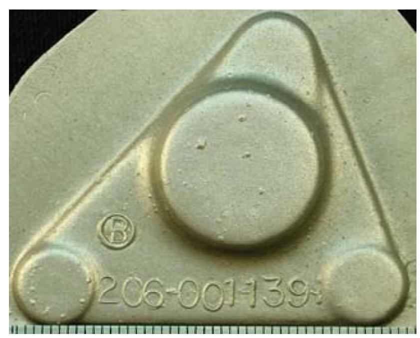


Figure 5: A plot of forging load vs. temperature for ZK60 alloy showing the effect of grain size on the forging load at various temperatures.



Coarse Grain

Fine Grain – 2



Fine Grain – 4

Figure 6: First trial forging at 650°F stock and 600°F die temperature using ultrafine-grained (UFG) forging stock produced by ECAE/P processing

Figure 7: Successful forging at 600°F from UFG AA 6061alloy stock produced by ECAE/P.

Figure 8: Same part (shown in Figure 7) successfully forged at 740°F using 15% less starting stock material showing reduced flash.

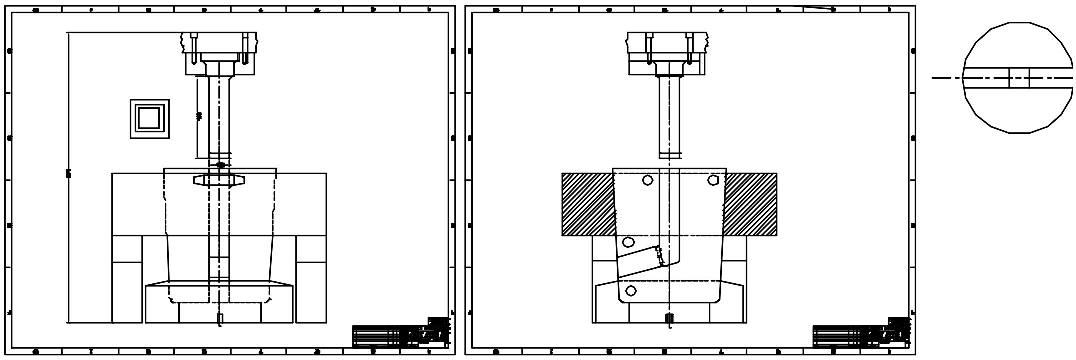


Figure 9: ECAE/P die for producing 100 mm x 100 mm cross section forging billets.

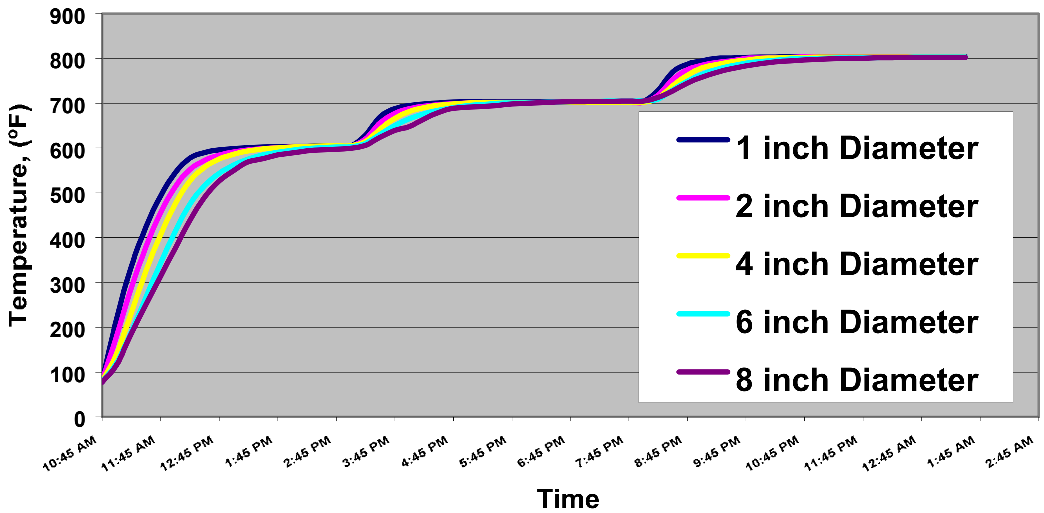


Figure 10: A plot of temperature vs. time for various sizes of forging stock in a typical forging stock heating furnace.

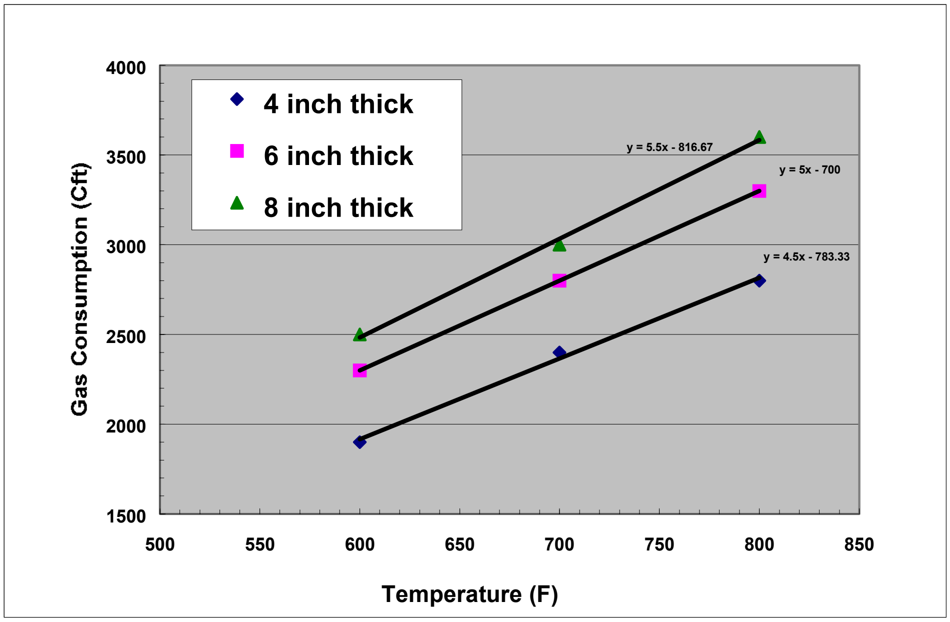


Figure 11: Gas consumption as a function of time to heat up for various sizes of forging stock in a typical forging stock heating furnace.

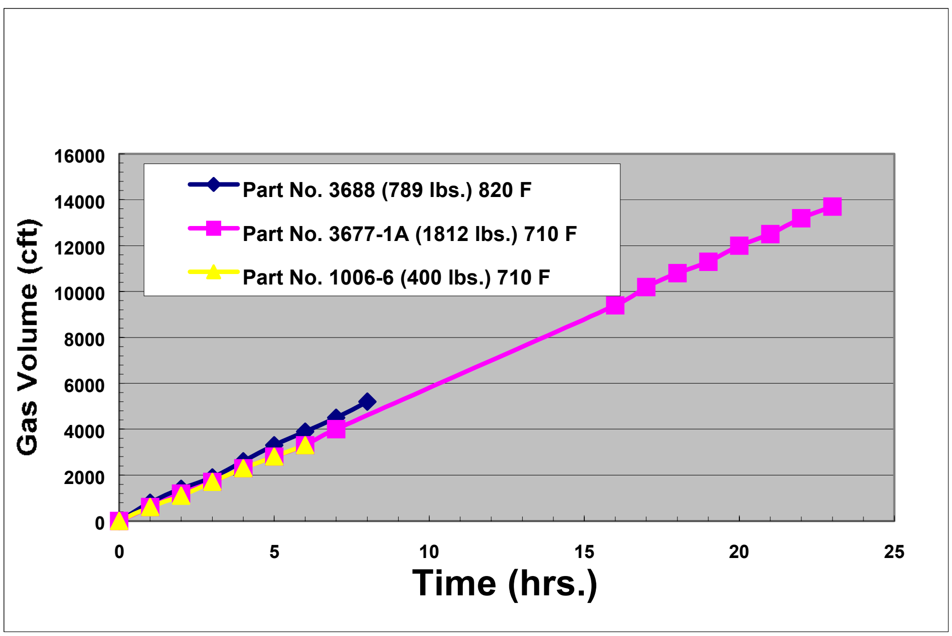


Figure 12: Gas consumption as a function of time for production loads of various size forgings in a typical forging stock heating furnace.

Plans for Next Year:

The project is on schedule on all tasks, except for those that are ORNL critical. There is a 4-5 months delay on these tasks because of the delay in release of funds to ORNL. We expect to reach Milestone 3 Fabrication and trials of the CSPD process during the first half of the second year. During the second year of the project we intend to continue working as per our original schedule, taking into consideration the delay that has already occurred.

Task 1. CSPD Process Development

a) Test 0.5-inch die - conduct CSPD processing trials on AA 6061, characterize material, validate computer modeling results, and document process variables and procedures.
b) Design and fabricate inserts for 1.125-inch cross-section material, and conduct CSPD processing trials on AA 6061, characterize material, validate computer modeling results, and document process variables and procedures.

Task 2. Demonstrate Energy and Cost Savings

a) Use redesigned die set to make 100-mm (4-inch) square UFG AA 6061material.
b) Conduct forging trials using 100 mm square material.
c) Conduct forging trials with CSPD material produced under Task 1, when material is available.
d) Refine energy and cost savings estimates for forging of UFG forging stock.

Task 3. Technology Transfer

Continue technology transfer activities as planned.

Patents:

The preliminary patent titled "Continuous Severe Plastic Deformation Process For Metals" is under development and will be filed during the first half of the second year. This is a comprehensive patent covering the technology. Additional patents on specific designs and of components and subcomponents of the designs will be filed at the appropriate time.

Table 4: Milestone Status Table

ID No.	***Description***	***Planned Completion***	***Actual Completion***	***Comments As of (2-20-02)***
Task 1	To demonstrate a CSPD process	12-31-03		
1.1	Design and build CSPD apparatus	3-31-02		Reviewed computer analyses. Anticipated delay due to delay in release of funds to ORNL
1.2	Produce UFG material by CSPD	12-31-03		
1.3	Make selected parts by forging	12-31-03		
Task 2	To demonstrate UFG material saves cost and energy during forging	3-31-03		
2.1	Prepare 50 mm ECAE material at AFRL	6-30-01	12-31-01	Completed
2.2	Characterize 50 mm ECAE material	6-30-01		On-going-delayed due to 2.1
2.3	Perform Forging with 50 mm ECAE material	6-30-02	1-31-02	Completed
2.4	Prepare 100 mm square ECAE material	3-31-03		On track
2.5	Perform forging with 100 mm ECAE material	3-31-03		On track
Task 3	To transfer technology to industry	3-31-04		On track
3.1	Identify additional commercial applications	12-31-02		On track
3.2	Conduct market assessment	12-31-02		On track
3.3	Conduct tech. transfer forums	12-31-03		On track
3.4	Exhibit CSPD technology at trade shows	12-31-03		On track
3.5	Seek commercialization partners	3-31-04		On track
Task 4	Reporting			On-track
4.1	Quarterly reports	12-31-03		Quarterly
4.2	Annual reports	5-20-03		90-days after anniversary
4.3	Final report	5-20-04		90-days after completion

Table 4: Milestone Status Table (cont.)

Milestone	*Description*	*Planned Completion*	*Actual Completion*	*Comments As of (2-20-02)*
1	Evaluate alternate CSPD designs and select trial designs	9-30-01		4-5 month delay due to delay in ORNL funds
2	Preliminary energy and cost calculations for using UFG material for forging	3-31-02		On track.
3	Complete fabrication of trial CSPD apparatus	3-31-02		4-5 month delay anticipated due to delay in ORNL funds
4	Produce CSPD samples of first alloy	6-30-02		May be delayed
5	Characterize and test CSPD samples of first alloy	12-31-02		On track
6	Demonstrate cost and energy savings forging fine grained material	3-31-03		On track
7	Evaluate and select optimum CSPD design and characterize samples	6-30-03		On track
8	Demonstrate cost and energy savings for forging CSPD material	12-31-03		On track

Budget Data (as of 2/20/02):

			Approved Spending Plan			Actual Spent to Date		
Phase / Budget Period			DOE Amount	Cost Share	Total	DOE Amount	Cost Share	Total
	From	To						
Year 1	2/21/01	2/20/02	$246,452	$439,126	$685,578	$109,468	$230,778	$340,246
Year 2	2/21/02	2/20/03	$229,224	$420,482	$649,706			
Year 3	2/21/03	2/20/04	$231,336	$393,968	$625,304			
Year 4								
Year 5								
		Totals	$707,012	$1,253,576	$1,960,588 (a)	$109,468	$230,778	$340,246 (b)

(a) Planned expenditures of $180,000 per year at ORNL are not included in this total
(b) ORNL expenditures of $45,152 over the period August 2001 – February 2002 are not included in this total

Spending Plan for 2nd Year (Feb 21, 2002 – Feb 20, 2003):

Month	Estimated Spending		
	WSU Contract	ORNL	Total
March, 2002	$17,040	$15,000	$32,040
April, 2002	$17,040	$15,000	$32,040
May, 2002	$17,040	$15,000	$32,040
June, 2002	$27,183	$15,000	$42,183
July, 2002	$22,607	$15,000	$37,607
August, 2002	$23,322	$15,000	$38,322
September, 2002	$16,969	$15,000	$31,969
October, 2002	$16,969	$15,000	$31,969
November, 2002	$18,399	$15,000	$33,399
December, 2002	$16,969	$15,000	$31,969
January, 2003	$17,755	$15,000	$32,755
February, 2003	$19,185	$15,000	$34,185
Totals	$230,477	$180,000	$410,477